Characterization of Waste Plastic Bag as a Novel Binder System and Homogeneity Test for Stainless Steel 316L Metal Injection Molding

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***Abstract*—This paper presents the characterization and mixing technique of the newly developed binder system that made up of palm kernel (PK) and waste plastic bag (source of High Density Polyethylene) to produce feedstock for green metal injection molding (MIM) applications. The thermal evaluation (TGA; DTA), FTIR analysis and density test were firstly performed to characterize the binders in purpose to design the overall features of the mixing process and subsequent processes of MIM. It is worth to know that mixing process is the paramount processes in the MIM technology since it will give the major effect to the following processes and even cannot be altered later. Hence, the appropriate mixing technique (mixing temperature, binder composition, powder loading, mixing speed) must be properly fixed in purpose to produce the homogeneous feedstock. Owing to this, the binder burnt-out test, density test and morphology study were made in order to investigate the homogeneity of the feedstock. The all conducted tests shown that the application of the waste plastic bag as a novel binder in MIM was acceptable, the mixing process was performed in a proper way by considering the characterization of the binders and the mixed feedstock shown the good homogeneity condition. These preliminary properties of the feedstock serve as a benchmark in designing the next schedule for the next whole steps (i.e. injection, debinding and sintering processes).**

Keyword-*High Density Polyethylene; homogeneity; metal injection molding; mixing; palm kernel*

I. INTRODUCTION

For many years, the development of a binder system is regularly being the major attentions among the researchers of MIM process. This effort has headed to the several improvements involving cost reduction, reducing environmental problems and much more. So far, many of the MIM researchers broadly explored the application of the natural resources binder such as paraffin wax, carnauba wax and such on. Nevertheless, only few of them are focusing on the waste resources of recycle products, i.e.; discarded tires, plastic bottles, etc. [1]. Related to this, authors focused on the waste disposal problems that are being crucial issues in the world due to the rapid development todays. The large tons of the waste plastic materials are produced daily and it has greater effect to the environment. Additionally, the plastic material can be classified as the non-biodegradable where it is disposed in the landfill, which is limited due to the high cost of incineration and limited space [2]. The waste plastic bag is one of the major plastic material sources for the daily used. Thus, this study is fundamentally to investigate the potential of using waste plastic bag (sometimes written as High Density Polyethylene), commonly abbreviated as a backbone binder. It is believes that such waste has a great potential which to be converted into the more beneficial industrial products.

In general, mixing process of MIM applications is the process where the all material are mixed in purpose to produce homogeneous feedstock [3]. The components includes are consist of the metal powder, primary binder (binder is removed during solvent debinding process) and secondary binder (binder is removed during thermal debinding process). It is worth to give the prior focused on the binder's characterization and feedstock preparation for MIM which is the crucial step since deficiencies in quality of the feedstock cannot be altered by subsequent processing modifications [4]. According to this, the binder's characterization should be well conducted for designing the features of mixing process to produce the homogeneous feedstock and free of powder-binder separation or particle segregation [3]. Failure in dispersing the powder uniformly in the binder and unsuitable rheological behavior of the feedstock will cause molding defects such as distortion, cracks, or voids which will lead to non-uniform shrinkage or warping of the sintered parts [3], [5].

II. Materials And Methods

A. *Metal powder*

Water atomized Stainless Steel 316L powder having irregular shape with mean size d^{50} = 6µm and supplied by Epson Atmix Japan was applied as the metal powder. According to Ibrahim et al (2009), the critical powder volume concentration (CPVC) for this metal powder found to be approximately 64.80 % [6]. According to German & Bose (1997), the optimum powder loading should be kept approximately 2-5% lower than critical loading [7]. Consequently, the powder loading applied for this MIM study is 60 vol. %. The metal powder characteristics are shown in Table I.

TABLE I. SS316L powder characteristic [6]

Characteristic	Details
Identification	SS316L, PF-10F
Tap density, g/cm^3	4.06
True Pcynometer density, /cm^3	8.0471
Powder size	d_{10}=2.87µm
Identification	d_{50}=5.96µm d_{90}=10.65µm

B. *Binders characterization*

Fourier Transform Infrared Spectroscopy (FTIR): The main rule in designing the new developed green binder system (not a commercial binders) of metal injection molding is to determine the major material contents in the selected recycled product (waste plastic bag). It is crucial to detect the existing of the major contents of High Density Polyethylene which is act as the secondary binder for this MIM study. Indeed, this analysis are being the most required testing that must be firstly conducted before proceeding to the other processes.

Density measurement: The Mettler Toledo XS 64 was used in determining the density of waste plastic bag and palm kernel via Archimedes principles. The density determination of the binders are very crucial in calculating the feedstock composition.

Thermal analysis: The thermal degradation of the binders were measured by Thermo Gravimetric Analysis (TGA) using the Linseis Thermo balance from 30 ºC to 900 ºC with heating rate of 10 ºC/min in nitrogen atmosphere. Whereby, the melting temperature of the binders were measured by Differential Thermal Analysis (DTA) from range of room temperature to 600 ºC with heating rate of 10 ºC/min. Knowing this properties are important as it is essential in designing an appropriate parameter of mixing process, injection process, debinding cycles and sintering process, so that the applied temperature during the aforementioned processes is within the exact range.

Homogeneity test: Homogeneity of the feedstock was measured by analyzed the density variation which applied the Archimedes water immersion method according to MPIF Standard 42, binder burn-out test using thermo gravimetric (TGA), and Field Emission Scanning Electron Microscope (FESEM).

III.RESULTS AND DISSCUSSION

A. *Characterization of binders*

For this present study, it is successfully claimed that the High Density Polyethylene (HDPE) was existed in the waste plastic bag. This is confirmed by two core reasons of FTIR analysis. The first reason is by considering the best hit score of the sample (waste plastic bag) was the Polyethylene High Density (HDPE) which valued by 0.926301. Table II shows the three of twenty search reference spectrum, the second rank of the detect spectrum is Polyamide Resin Melting PT95Deg (0.879219) whereby the last rank of the spectrum is Styrene/Butadiene Copolymer ABA Copolymer 30% Styrene which valued 0.398797. Whereby, the second reason was by comparing the spectra of the waste plastic bag (sample) with the original spectra of search library with name of Polyethylene High Density by code AP0049. Consequently, fig.1 shown the spectrum pattern of the sample and original spectra of HDPE are likely the same. Thus, this two core reasons revealed the existing of the major contains of HDPE in the waste plastic bag (WPB). Evidently, the waste plastic bag is qualified to be applied in this study as a green binders which is act as the source of HDPE.

TABLE III. Searched references result from FTIR analysis

Search score	Search reference spectrum description
0.926301	Polyethylene High Density
0.879219	Polyamide Resin Melting PT95Deg
0.398797	Styrene/Butadiene Copolymer ABA Copolymer 30% Styrene

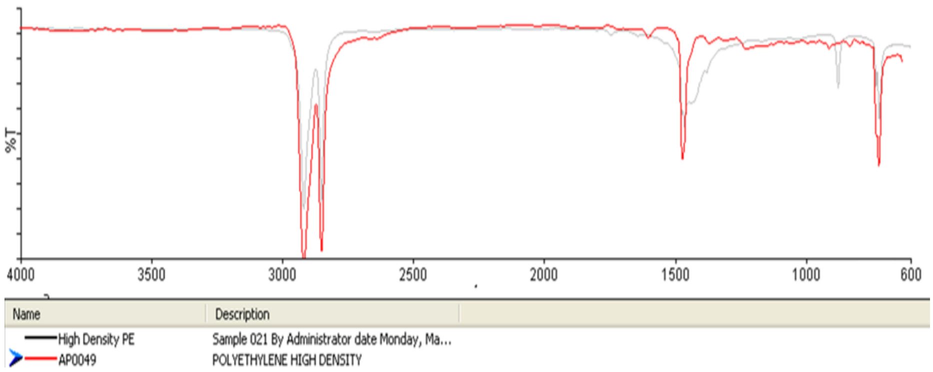
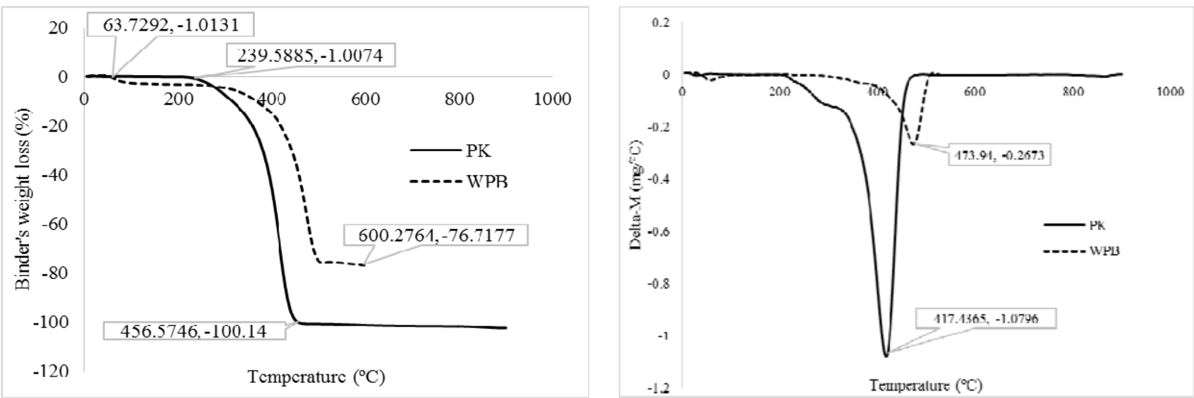


Fig. 1. Spectra of sample (waste plastic bag/High Density PE) and library search (POLYETHYLENE HIGH DENSITY/ AP0049)

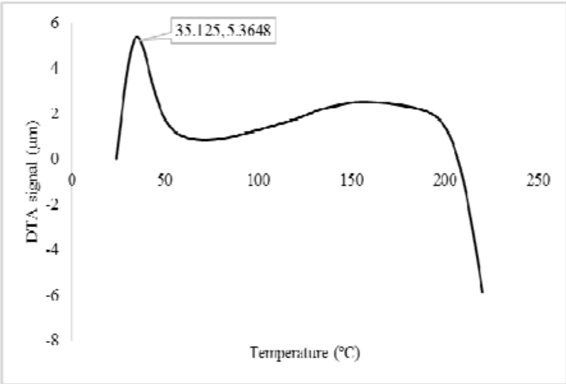
The Thermo Gravimetric Analyzer (TGA) was utilized to examine the thermal degradation of the binders. As displayed in Fig. 2 (a) and (b) are the TGA curve for both binders which are the palm kernel (PK) and waste plastic bag (WPB). Referring Fig. 2 (a), palm kernel starts to degrade at 63.7292 ºC (weight loss of PK is 1.0131%) and finish at 456.5746 ºC (weight loss of PK is 100.14%). Whereas, waste plastic bag degraded at the range temperature of 239.5885 ºC (weight loss of waste plastic bag is 1.0074%) to 600.2764 ºC (weight loss of waste plastic bag is 76.7177%). Details in that, Fig. 2 (b) focusing on the optimum degradation temperature of the binders. Palm kernel is best degraded at 417.4365 ºC whereby the waste plastic bag is maximum degraded at 473.94 ºC. Consequently, these two binders are considered having the wide range of degradation temperature. Thus, this wide degradation temperature is very useful for a fast debinding process [4],[8], a defect-free product [4],[9] and facilitates shape retention [8],[10]. Apart from that, TGA curve was also used to design the thermal debinding cycle [9], where all binders were removed at the above maximum degradation temperature of the binders. Other than that, the temperature should not be raised too quickly in order to prevent defects, such as bubbles and cracks.



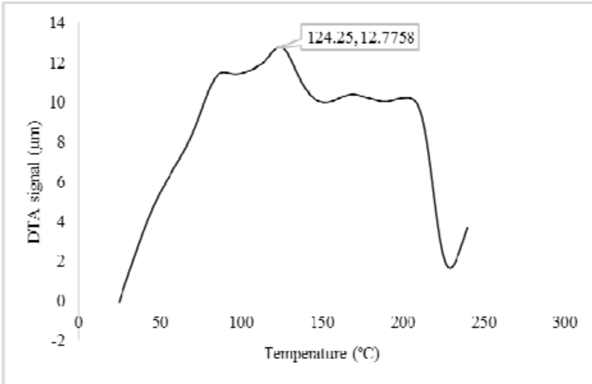
(a) (b)

Fig. 2. (a) Degradation graph of palm kernel and waste plastic bag, (b) Optimum degradation graph of palm kernel and waste plastic bag

Whereas, the peak of the DTA graph is corresponding to the melting temperature of the binder component. Fig. 3 (a), it shows the palm kernel is optimally melts at 35.125 ºC. From Fig. 3 (b), it shown that melting temperature of the waste plastic bag (HDPE) was originally at 124.25 ºC. In that sense, the mixing temperature should be higher than 124.25 ºC. This is supported by German and Bose (1997) that proved the mixing temperature should be higher than the highest melting temperature of binders and lower than the lowest degradation temperature of the binders [7]. Besides that, the density of the waste plastic bag and palm kernel are 1.0548 g/cm^3 and 0.8852 g/cm^3 respectively which are shown in the Table III. This result is very significant to calculate the feedstock composition. Also, the summary of the characterization of the binders (TGA, DTA, density) and the binder composition of this metal injection molding are listed in Table III.



(a)



(b)

Fig. 3. (a) Melting graph of palm kernel, (b) Melting graph of waste plastic bag

Table IV shows the properties of the commercial HDPE which is conducted from the previous studies [10]-[12]. Evidently, the waste plastic bag (HDPE) is having the great similarities to theoretical properties of the commercial HDPE. The results of the waste plastic bag characterization were compared with the properties of commercial HDPE in order to ensure that the finding results is not different too much compared to the theoretical values. Then, this present study have shown that the melting temperature and degradation temperature of the waste plastic bag (as source of HDPE) are lies between the range of the degradation temperature (462 ºC to 550 ºC) and melting temperature (122 ºC to 132 ºC) of the commercial HDPE as shown in Table IV . However, the density of waste plastic bag is slightly different from the theoretical density of commercial HDPE which the differences is valued by 0.0818 g/cm^3. By comparing of this, authors claimed that it is acceptable to use the waste plastic bag which mainly purpose to apply “Green MIM” and reduce manufacturing cost instead of using the commercial HDPE.

TABLE IIIII. Summary of the binder’s characterization

Binders	Palm kernel	Waste plastic bag
Binder composition (wt. %)	60	40
Degradation temperature (ºC)	417.4365	473.94
Melting temperature (ºC)	35.125	124.25
Density (g/cm^3)	0.8852	1.0548

TABLE IV. The properties of commercial HDPE

Density (g/cm^3)	Degradation temperature (ºC)	Melting temperature (ºC)	Ref.
0.95 – 0.973	462 – 550	122 - 132	[10],[11],[12]

Continuity from this, the homogeneous feedstock should be produced. According to German and Bose (1997), there are several factors such as time, temperature, powder size and shape, formulation of binder, shear rate, and powder loading are parameters that need to be considered [7]. Nevertheless, in this study, only three parameters were selected in order to establish a suitable mixing condition. These were mixing temperature, mixing speed and mixing time. Detailing in this, German and Bose (1997) also stated that molding and mixing temperatures must be below the binder decomposition to prevent binder degradation and higher than the highest melting point of the binders in order to ensure the binders is completely melt, and flow ability of the molten feedstock during molding process [7]. By considering this statement, the mixing temperature that fixed for this study should be higher than 124.25 ºC. However, 10 ºC is added due to the mixing machine errors. Thus, the mixing temperature of 135ºC was selected to prevent the binder constituent from degrade since it is within the highest melting temperature (124.8ºC) and the lowest degradation temperature of the binder system (417.5ºC), thus allowing complete melting of palm kernel and waste plastic bag.

(a) (b)

Fig. 4. (a) Manual extracting of waste plastic bag, (b) Crushed feedstock

Fig. 4 (a) and (b) shows the manually trimming of waste plastic bag and the crushed feedstock. The composition binder of PK/HDPE (60/40) were mixed with stainless steel powder 316L at the temperature 135ºC by using a rotary mixed (Brabender Plastograph EC) for 80 minutes. Mixing speed was kept constantly at 30 rpm. Waste plastic bag was heated for about 10 minutes followed by the addition of metal powder step by step until all the metal powder mixed well with melt waste plastic bag. After 20 minutes, palm kernel was added into the mixer and blended with the rest of the compositions for about 50 minutes. Later, the blended feedstock is taken out from the mixer and leave to cool at room temperature before being crushed into small pallet using Granulator machine. Fig. 4 (b) shown the crushed feedstock by using the Plastic Granulator SLM 50FY machine.

B. CHARACTERIZATION OF FEEDSTOCK

The main goal of mixing is to produce a homogeneous feedstock. The homogeneity of the feedstock was examined by binder burn-out test, density test and Field Emission Scanning Electron (FESEM).

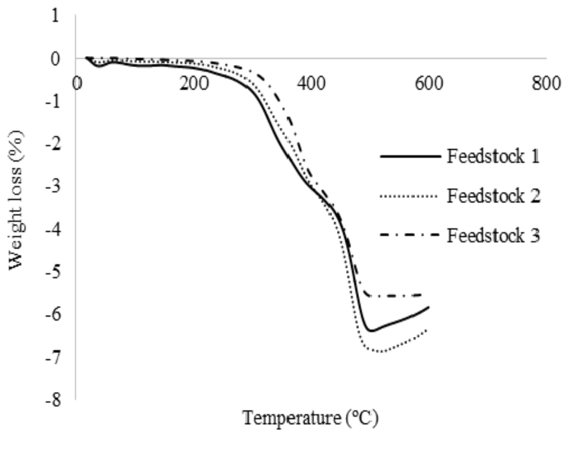


Fig. 5. TGA curve for 3 pieces of same batch of mixing feedstock

For the binder burnt-out test, the homogeneity of the feedstock was assessed by comparing the weight loss (at the end of the testing) of three different samples for same batch of mixing session. The samples that have the approximately same weight is chosen for testing on a Perkin-Elmer TGA model 7. The heating in nitrogen atmosphere was conducted from 30 ºC to 600 ºC using a rate of 10 ºC/min. The weight loss percentage of the binders can be found through thermo gravimetric curves that represent the percentage of the binder loss when heating in nitrogen atmosphere. Displayed in Fig. 5 are the three TGA curve that are made up of the three samples of same batch mixing feedstock. This combination graph shown the well replicated of TGA curve which indicates a good homogeneity of feedstock. Whereby, Table V details shown the binder's weight loss of feedstock. It can be observed that there are slightly differences in the values, but it is still in acceptable range of homogeneity feedstock

TABLE V. Weight loss for binder of several samples

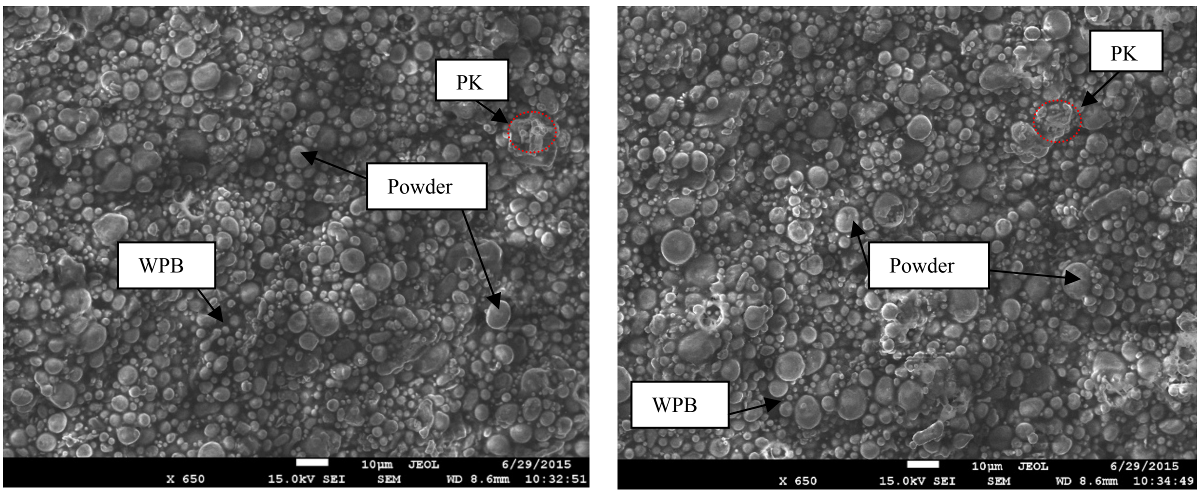
Sample	Weight loss (%)
1	5.8
2	6.3
3	5.5

Amin et al (2014) claimed that the higher powder loading leads to increase feedstock density [13]. Also, an inhomogeneous feedstock can result in density gradients within the molded part and cause a distortion [3]. One way to evaluate the feedstock homogeneity includes sampling the feedstock density from various portions of the same batch. The results of the density measurement for feedstock of five different samples are shown in Table VI. It can be observed that there is slight difference of density value. However, due to the small differences, it was considered obtaining a good feedstock homogeneity.

TABLE VI. Density data for homogeneity test

Feedstock	Average density
Sample 1	4.7216
Sample 2	4.7849
Sample 3	4.7650
Sample 4	4.7715
Sample 5	4.8211

Microstructures of the feedstock was evaluated by Field Emission Scanning Electron Micrographs. Shown in Fig. 6 (a) and (b) are the microstructure of the feedstock. The microstructure reveals that there are reasonably uniform distribution of metal particles, waste plastic bag (WPB) and palm kernel (PK). Stainless steel and binder system could be distinguished due to various contrast levels caused by different levels of atomic number [14]. Stainless steel appears brighter than the binder system due to more back-scattered electrons released by virtue of its higher atomic number [14]. The metal particles phase is shown as brighter phase, while the HDPE phase is dark (black). Whereby, the palm kernel particles are embedded within the amorphous matrix composed of randomly distributed in the matrix planar boundaries. The contact surface powder with binder was good because the particle were coated by binder. There was no powder-binder separation phenomenon occurred in this feedstock because of the good contact surface between binder and powder. Good surface contact between binders with powder will make sure metal particle will hold tightly and prevent cracked or damage in the further processes.



(a) (b)

Fig. 6. Field Emission Scanning Electron Micrograph (FESEM) structure of the feedstock

IV. CONCLUSION

The authors claimed that the waste plastic bag is acceptable to be applied as a novel binder in this MIM technology instead of using the commercial HDPE which is quite expensive. This is revealed by the existing of the major contents of the HDPE as a part of the waste plastic bag. Meanwhile, the density of the waste plastic bag is approximately same with the theoretical value of the commercial HDPE which having slightly difference by 0.0818 g/cm^3. Indeed, the calculation of the feedstock formulation was made based on the binder's densities. Whereby, by considering the degradation temperature and melting temperature of the binders, the mixing temperature is fixed at 135 ºC which is ideal temperature where the binders are completely melt and bind the metal powder. By considering the homogeneity test and morphological study that were made for the feedstock, the authors claimed that the feedstock was having good homogeneity and the further processes should be conducted by applying this mixing technique and formulation.

ACKNOWLEDGMENT

Our deepest appreciation to Advanced Manufacturing and Material Center (AMMC), Universiti Tun Hussein Onn Malaysia for research facilities and support.

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